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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant	:	Michael A. Todd	Group Art Unit 2814
Appl. No.	:	10/074,534	
Filed	:	February 11, 2002	
For	:	PROCESS FOR DEPOSITION OF SEMICONDUCTOR FILMS	
Examiner	:	Shrinivas H. Rao	

DECLARATION OF DR. MICHAEL A. TODD UNDER 37 C.F.R. § 1.132

I, Michael A. Todd, declare that:

1. I have the B.S. degree in Physics (1991) and the Ph.D. in Chemistry (1996) from Arizona State University. My Ph.D. research was directed to chemical vapor deposition ("CVD") processes. Most of my professional career since graduation has focused on the research and development of CVD processes for semiconductor fabrication applications. I am a named inventor on at least five U.S. patents and an author on numerous technical publications in these areas. I consider myself skilled in the art of CVD processes for semiconductor fabrication applications.

2. I am familiar with the course of prosecution of the above-captioned patent application, including the Office Action mailed on March 25, 2004, in which the "Rolfson" (U.S. Patent No. 5,786,027) and "U'Ren" (U.S. Patent No. 6,365,479) references were discussed.

3. I have carefully studied the Rolfson reference. The "Background of the Invention" section of Rolfson explains that polycrystalline silicon has a microstructure that includes grains of crystalline silicon that are separated from one another by grain boundaries, *see* Rolfson at 1:34-42 (column:lines). Rolfson explains that the grain boundaries present a number of challenges, *see* Rolfson at 1:43-2:4. In particular, Rolfson explains that grain boundaries that are continuous in

the z-direction through the full thickness of the film are a particular problem because a wet chemical can strip the oxidized grain boundaries leaving the underlying substrate exposed, *see* Rolfson at 2:5-17. Such continuous grain boundaries can also affect the smoothness and structural integrity of the film, *see* Rolfson at 2:18-26.

4. Rolfson Figure 1, reproduced below, shows a polysilicon film 10 deposited in accordance with a prior art low pressure CVD process, *see* Rolfson at 3:23-25. Grain boundaries 16 are continuous in the z-direction and extend through the full thickness of the polysilcon layer 10, *see* Rolfson at 3:40-45. Rolfson explains that wet chemicals can strip the grain boundaries, causing the substrate 12 to be attacked and damaged, *see* Rolfson at 3:46-54. The process for depositing such prior art films may include a deposition temperature of 675° C, *see* Rolfson at 3:32-39; *see also* 1:25-32.

Rolfson Figure 1

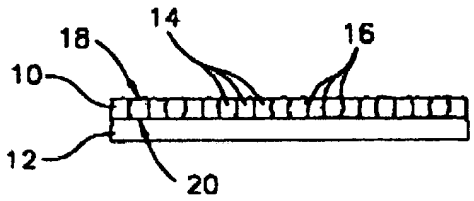


FIGURE 1
(PRIOR ART)

5. Rolfson is directed to a low pressure CVD process in which at least two silicon source gases having different adsorption characteristics (termed "sticking coefficients") are used to grow a polysilicon thin film on a substrate, *see* Rolfson at 2:43-49. The reason for using two different silicon source gases is to form a polycrystalline film with grain boundaries that are not continuous across the full thickness of the film, *see* Rolfson at 2:51-56 and 4:24-33. Rolfson Figure 3, reproduced below, illustrates a polysilicon film with discontinuous and randomly oriented grain boundaries deposited by the allegedly inventive method, see Rolfson at 3:55-58 and 4:55 to 5:2. With reference to Figure 3, Rolfson explains that:

> The polysilicon layer 10A includes grains 14A and grain boundaries 16A. The grain boundaries 16A are not continuous in the z-direction from the surface 18A of the polysilicon layer 10A to the interface 20A with the substrate 12. Rather, the grain boundaries 16A are discontinuous across the thickness of the polysilicon layer 10A. In addition, the grain boundaries 16A do not follow a particular

orientation of the fiber axes of the polysilicon layer 10A. Rather, the grain boundaries 16A are randomly oriented with respect to the {110} and {100} fiber axes. With a random grain orientation, in a subsequent process, such as cleaning or wet etching, it is less likely the (sic) that the substrate 12 will be left exposed by stripping of the grain boundaries 16A.

Rolfson Figure 3

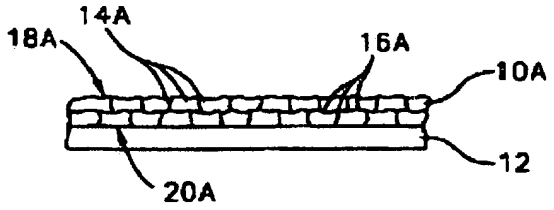


FIGURE 3

6. Considered as a whole, Rolfson teaches the undesirability of film surface roughness and grain boundaries that extend from the surface of the film to the underlying substrate. Thus, Rolfson teaches the undesirability of the prior art film shown in Rolfson Figure 1 and the processes used to form it, including a deposition temperature of 675° C.

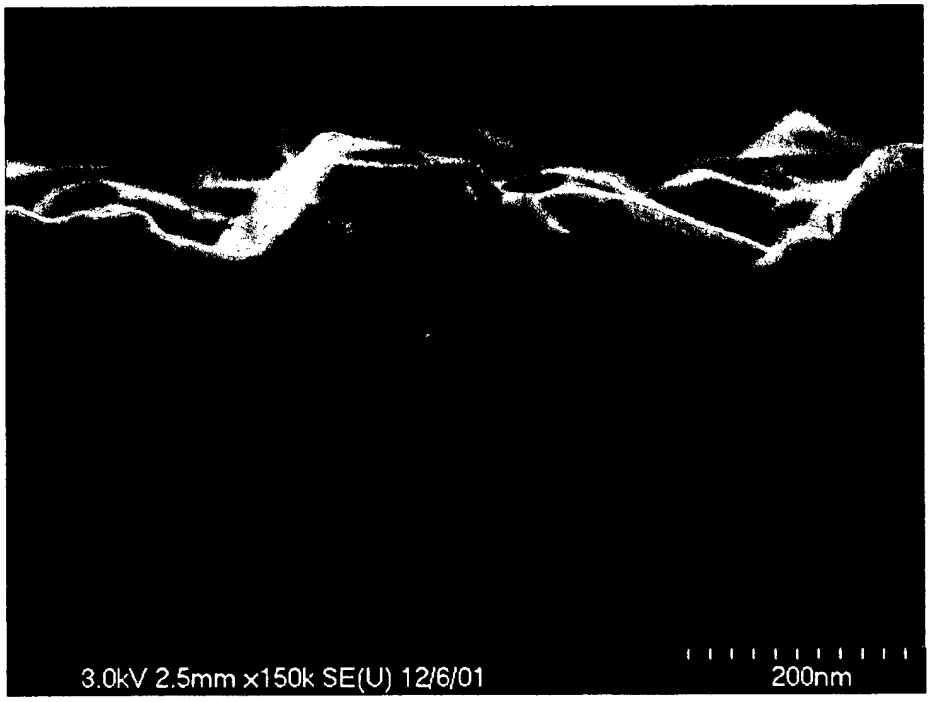
7. I have carefully studied the U'Ren reference. U'Ren discloses methods for depositing silicon-germanium ("SiGe") films for use in semiconductor devices, *see* U'Ren at 1:9-11. U'Ren purports to disclose a method for concurrently depositing a polycrystalline SiGe region and a single-crystal SiGe region, *see* U'Ren at 2:57-62. U'Ren discloses that such concurrent deposition is accomplished by using a deposition temperature and pressure at which deposition of polycrystalline SiGe occurs in a mass-controlled mode and deposition of single crystal SiGe occurs in a kinetically-controlled mode, *see* U'Ren at 2:57-62; Figure 3; 7:19 to 10:57. U'Ren indicates that such concurrent deposition imposes significant constraints on the deposition temperature and pressure, *see* U'Ren Figure 3. For example, U'Ren sets a lower limit on the operating temperature of 650° C at 100 Torr in order to achieve more polycrystalline than amorphous growth, *see* U'Ren at 10:10-26. U'Ren indicates that lower temperatures and pressures are undesirable because there is more amorphous deposition, *see* U'Ren at 10:10-26.

8. One skilled in the art, at the time of the invention claimed in the above-captioned patent application, would not have been motivated by the disclosure of U'Ren to include a germanium source in the Rolfson CVD process because it was generally believed that the presence of germanium resulted in increased surface roughness, larger average grain size, and

increased formation of grain boundaries that extend from the surface of the film to the underlying substrate. For example, those skilled in the art were generally aware that the addition of germane to a silane-based CVD process increased the roughness and the depth of the grain boundaries in the resulting SiGe film, *see* V. Z-Q Li *et al.*, Appl. Phys. Lett. 71(23) 3388-90 (1997) (attached as Exhibit A); T-J King *et al.*, J. Electrochem. Soc., 141(8) 2235-41 (1994) (attached as Exhibit B); A. Kovalgin *et al.*, ProRISC/IEEE 311-17 (1998) (attached as Exhibit C); C. Hernandez *et al.*, Mat. Res. Soc. Symp. Proc. 533 93-98 (1998) (attached as Exhibit D).

9. The effect of including germanium is also illustrated in Figure 13 of the above-captioned patent application, reproduced below, which shows an SEM photomicrograph of a cross-sectioned sample of a SiGe film deposited onto a substrate using silane and germane:

Figure 13



10. As discussed in the text of the above-captioned patent application at paragraph [0105], the SiGe film shown in Figure 13 has a surface roughness of 226 Å. The surface shown is so rough that some of the grain boundaries extend from the surface of the film to the underlying substrate. The degree of roughness and the extent to which the grain boundaries extend from the surface of the film to the underlying substrate shown in Figure 13 are inconsistent with Rolfson's

goal, *see* Rolfson Figure 3. The effect illustrated in Figure 13 is consistent with the reported literature, *see* Exhibit A (Fig.1) and Exhibit B (Fig. 11).

11. As noted in paragraph 5 above, Rolfson is directed to a low pressure CVD process in which at least two silicon source gases having different adsorption characteristics (termed "sticking coefficients") are used to grow a polysilicon thin film on a substrate, *see* Rolfson at 2:43-49. The reason for using two different silicon source gases with different sticking coefficients is to form a polycrystalline film with grain boundaries that are not continuous across the full thickness of the film, *see* Rolfson at 2:51-56. Rolfson explains at 4:24-33 that, by using multiple silicon source gases with different sticking coefficients, a more random surface condition is present during the deposition process, leading to grains forming with discontinuous and randomly oriented grain boundaries. Thus, the Rolfson CVD process favors the use of silicon sources having sticking coefficients with relatively large differences from one another.

12. Those skilled in the art, at the time of the invention claimed in the above-captioned patent application, understood that the relative difference in sticking coefficient between one silicon source and another was effectively reduced when the silicon sources were in the presence of a germanium source in a CVD process, *see* M. Cao *et al.*, J. Electrochem. Soc., 142(5) 1566-72 (1995) (attached as Exhibit E) and J. Holleman *et al.*, J. Electrochem. Soc. 140(6) 1717-22 (1993) (attached as Exhibit F).

13. One skilled in the art, at the time of the invention claimed in the above-captioned patent application, would not have been motivated by the disclosure of U'Ren to include a germanium source in the Rolfson CVD process because one skilled in the art would have believed that the presence of the germanium source would reduce the effective difference in sticking coefficients between the silicon sources, thereby reducing the effectiveness of the method employed by Rolfson to achieve discontinuous and randomly oriented grain boundaries.

14. As noted in paragraph 5 above, Rolfson is directed to a low pressure CVD process in which at least two silicon source gases having different adsorption characteristics are used to grow a polysilicon thin film on a substrate, *see* Rolfson at 2:43-49. Rolfson states that the low pressure CVD process can be carried out in a standard low pressure CVD furnace at temperatures of from 580° C to 650° C and at pressures of from about 200 mTorr to 1 Torr, *see* Rolfson at 4:6-8.

15. The aforementioned Office Action at page 8 refers to a passage in Rolfson at 3:38-40 and states that it "describes temperature up to 675 degrees." However, the context of that Rolfson passage is the prior art shown in Rolfson Figure 1, which Rolfson teaches is undesirable because it can cause the substrate to be attacked and damaged by the wet chemical, *see* Rolfson at 3:53-54. Therefore, when considered as a whole at the time of the invention claimed in the above-captioned patent application, one skilled in the art would not have been motivated by the disclosure of U'Ren to utilize a deposition temperature of 675° C in the Rolfson CVD process because Rolfson associates this temperature with a process that produces an undesired result.

16. As noted in paragraph 14 above, Rolfson states that the low pressure CVD process can be carried out in a standard low pressure CVD furnace. Those skilled in the art at the time of the invention claimed in the above-captioned patent application generally recognized that a standard low pressure CVD furnace typically contained numerous wafers.

17. Those skilled in the art at the time of the invention claimed in the above-captioned patent application were aware of a generally undesirable phenomenon known as "depletion," *see* Exhibit F; *see also* Exhibit C. Depletion occurs during CVD processes conducted under mass controlled conditions in which the deposition rate is faster than the rate at which the reactant gas is supplied, resulting in non-uniform deposition across the surface of the substrate and/or between different substrates in a multiple substrate chamber. Depletion effects are exacerbated by the presence of germanium sources, *see* Exhibit F (e.g., p. 1719). Film composition plays an important role with regard to film texture and morphology, *see* Exhibit C (e.g., p. 311). Depletion is a particular problem in standard low pressure CVD furnaces because it results in wafer-to-wafer non-uniformities in film composition and film thickness, thereby leading to different grain structures in each of the films deposited in a given batch of wafers.

18. The depletion effects described in Exhibit C were observed at deposition temperatures well below the 580° C to 650° C range of Rolfson, *see* Exhibit C, Figs. 1-3 (430° C). One of ordinary skill in the art would have expected even greater depletion effects at the temperatures utilized by Rolfson, *see* Exhibit C (p. 312) and Exhibit F (Figure 3).

19. One skilled in the art, at the time of the invention claimed in the above-captioned patent application, would not have been motivated by the disclosure of U'Ren to include a germanium source in the Rolfson CVD process because one skilled in the art would have believed that the use of the germanium source under the U'Ren mass-controlled flow conditions would

have produced undesirable depletion effects in the standard low pressure CVD furnace utilized by Rolfson, particularly at the upper end of the 580° to 650° temperature range used by Rolfson.

20. One skilled in the art, at the time of the invention claimed in the above-captioned patent application, would not have had a reasonable expectation that the process of Rolfson could be successfully modified in light of U'Ren because the use of germanium and/or mass controlled deposition conditions would have caused significant depletion problems in the standard low pressure CVD furnaces used by Rolfson.

21. Rolfson discloses a deposition pressure/temperature combination of about 200 mTorr to 1 Torr and 580° C to 650° C, *see* Rolfson at 4:6-8. U'Ren discloses a deposition pressure/temperature combination of about 100 Torr to about 200 Torr at 650° C or higher, and about 650° C at 200 Torr, *see* U'Ren at 10:10-25. At lower pressures (75 Torr), U'Ren indicates that deposition becomes undesirably amorphous, *see* U'Ren at 10:10-25.

22. One skilled in the art, at the time of the invention claimed in the above-captioned patent application, would not have had a reasonable expectation that the process of Rolfson could be successfully modified in light of U'Ren because of the significant differences in the deposition pressure/temperature combinations disclosed by the two references. In particular, one skilled in the art would have believed that U'Ren's goal of achieving more polycrystalline than amorphous growth would be frustrated at the significantly different deposition pressure/temperature combination disclosed in Rolfson, and that Rolfson's goal of achieving randomly oriented grains by using multiple silicon sources with different sticking coefficients would be frustrated at the significantly different deposition pressure/temperature combination disclosed in U'Ren.

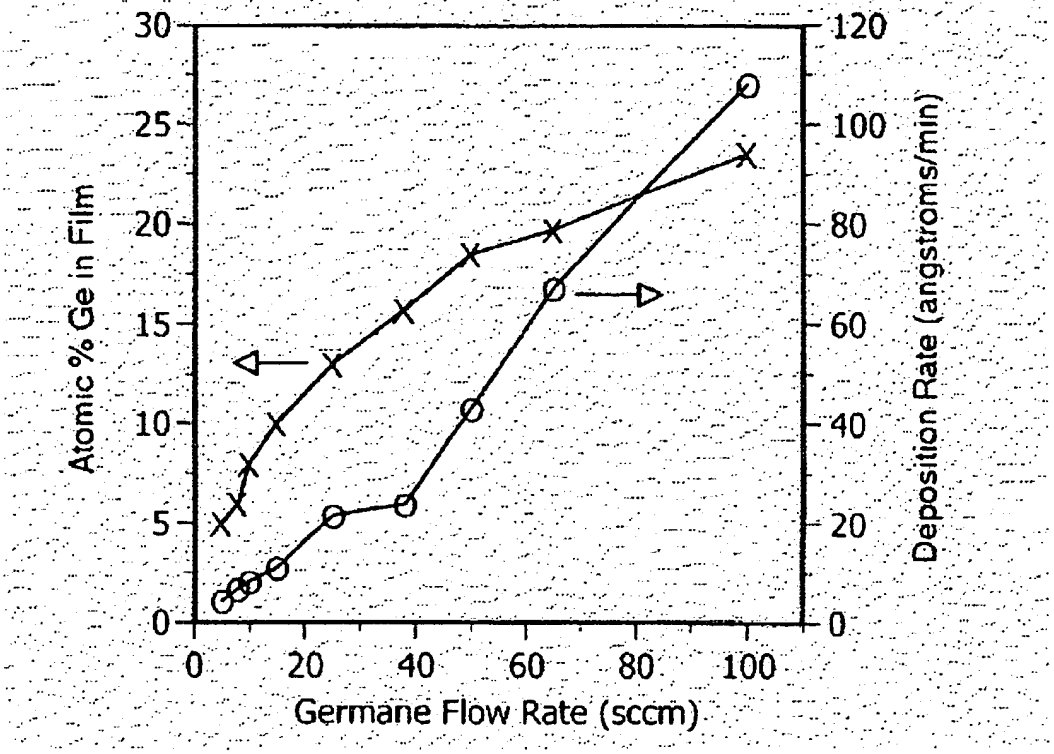
23. The aforementioned Office Action states at page 8 that "it is well known in the art that the LCVD (sic) AND RPCVD steps(having different pressures) (sic) have interchangeably used (sic) frequently in the prior art. . . . Rolfson's LPCVD and U'Ren's RPCVD steps have been interchangeably used in the prior art and therefore are compatable (sic) with each other." I disagree because purported interchangeability in certain processes does not necessarily imply interchangeability in all processes. With respect to the deposition of SiGe in particular, those skilled in the art understand that LPCVD and RPCVD are not *per se* interchangeable. For example, those skilled in the art consider the SiGe deposition results obtained under the conditions described in Exhibit F to be contradictory to those reported in the literature for LPCVD and APCVD systems, *see* Exhibit F (p. 1719). Thus, one skilled in the art would not

regard Rolfson's LPCVD and U'Ren's RPCVD steps to be interchangeable in the manner described in the aforementioned Office Action.

24. The aforementioned Office Action indicates at page 9 that Figures 5-10 and paragraphs 0052 to 0058 of the above-captioned patent application do not show unexpected results because "firstly the unexpected results are not set out and cannot be properly responded to, secondly the results described in the specification are the results that flow logically from the teachings of the prior art." I disagree with these statements for the reasons discussed below.

25. As explained in paragraphs 0053 to 0055, a series of SiGe films were deposited at various temperatures using a conventional silicon source (silane) and a germanium source (germane). However, the amount of germanium incorporated into the resulting films was not a linear function of the amount of germane in the deposition gas, as shown in Figures 5-8. Thus, as explained in paragraph 0053, Figure 5 shows that the amount of germanium incorporated into the resulting film (left-hand axis) is not a linear function of the amount of germane in the deposition gas. Figure 5 is reproduced below for the convenience of the Office. Figures 6-8 show that the non-linear behavior is observed at various temperatures and thus at various deposition rates, greatly complicating the task of depositing a smoothly graded SiGe film having a specified thickness and a specified Ge content because of the difficulties associated with simultaneously compensating for the non-linearities in both Ge concentration and film deposition rate.

Figure 5: Film Composition and Deposition Rate as a Function of Germane Flow Rate Using Silane at 600°C

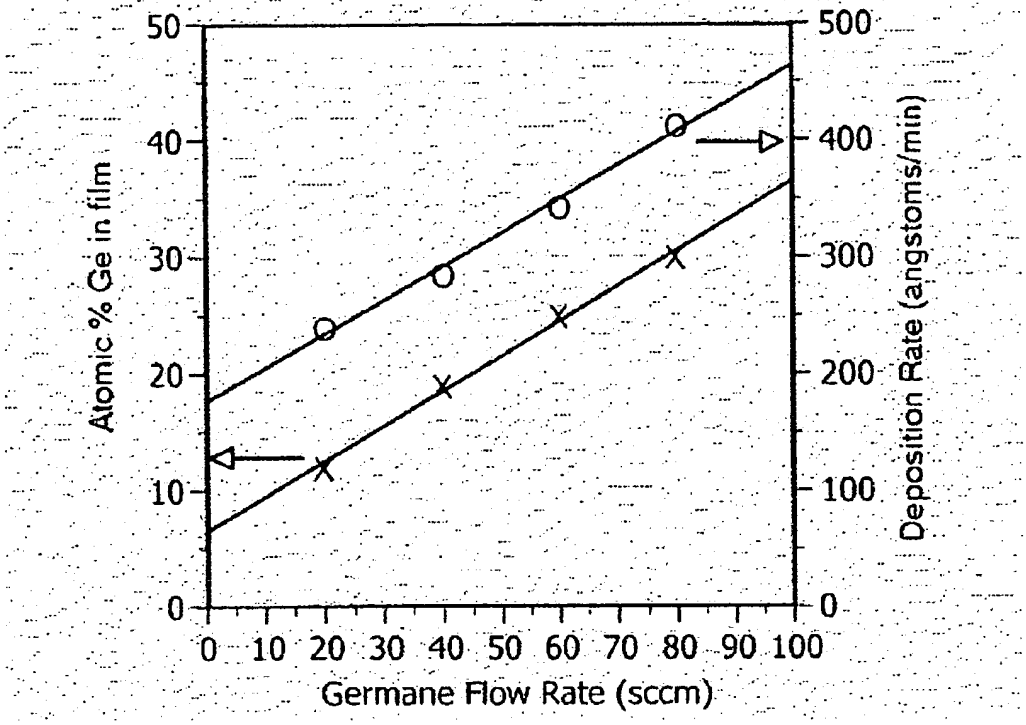


26. The substantial non-linearity of Ge incorporation over a broad range of Ge concentrations as shown in Figures 5-8 is recognized as a long-standing problem in the art, *see* Exhibit A (Fig. 2); Exhibit C (Fig. 1); Exhibit E (Fig. 9) and Exhibit F (Fig. 4). For example, Exhibit A at page 3389, column 1, last paragraph, notes that the germanium concentration in the SiGe film is not a linear function of the germane concentration in the gas phase when using either silane or disilane as the silicon source.

27. As explained in paragraph 0056, the unexpected effect of changing the amount of Ge precursor during CVD deposition using a trisilane-containing gas is shown in Figures 9-10. **In contrast to the non-linearities apparent in Figures 5-8, Figure 9 shows that the Ge incorporation into the film is, surprisingly, a substantially linear function of the germane flow rate when using trisilane as a silicon source.** Figure 9 is reproduced below for the convenience of the Office. Figure 10 also illustrates preferred substantial linearity of Ge incorporation and deposition rate for trisilane/germane under different conditions than illustrated

in Figure 9, demonstrating that the advantages of using trisilane are not limited to the specific conditions used to obtain the data in Figure 9.

Figure 9: Film Composition and Deposition Rate as a Function of Germane Flow Rate Using Trisilane at 600°C (H_2 Flow Rate = 20 slm)



28. The effect of trisilane on germanium incorporation and flow rate is highly unexpected in view of the long-standing art-recognized problem of non-linear Ge incorporation when using germane with traditional silicon precursors such as silane. Thus, the use of a deposition gas that contains trisilane greatly simplifies the task of depositing a graded Si-containing film using thermal CVD because such use of trisilane facilitates substantial linearity of Ge incorporation and deposition rate.

29. The above-captioned patent application also enables the use of trisilane to provide additional unexpected results such as higher deposition rates, higher degrees of film uniformity, and smoother surfaces, *see* paragraphs [0057] to [0058]. For example, Figure 12 (reproduced below) illustrates the relatively rough SiGe surface obtained using silane, whereas Figure 14 (reproduced below, same magnification and tilt angle as Figure 12) illustrates the much smoother surface obtained using trisilane, *see* Comparative Example 88 and Example 89.

Figure 12 (Silane)

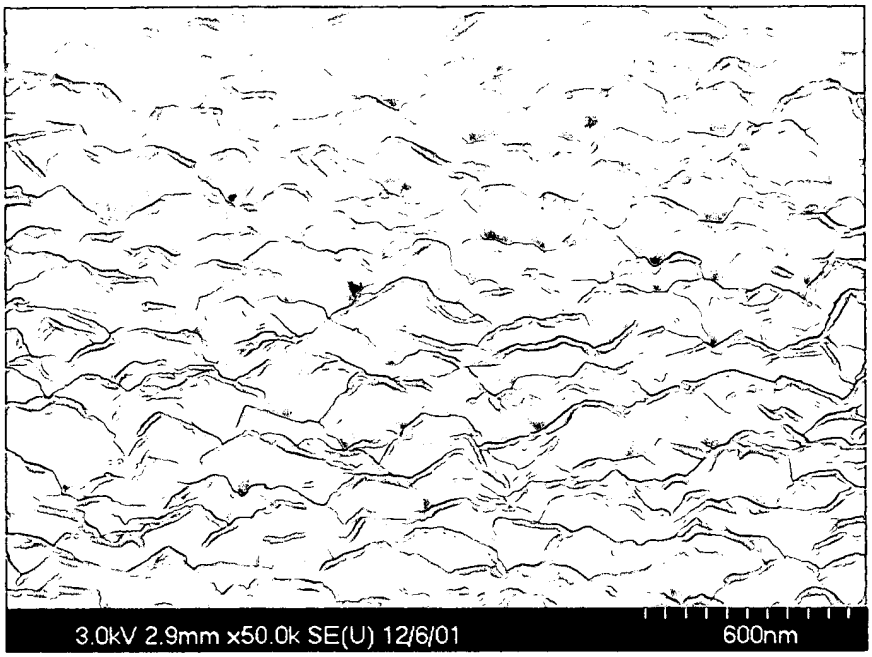


Figure 14 (Trisilane)

30. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued therefrom.

Dated: 23 June, 2004 By: Michael A. Todd
Michael A. Todd

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